

N° 1925



A.D. 1910

(Under International Convention.)

Date claimed for Patent under Patents and Designs  
Act, 1907, being date of first Foreign Application (in Germany), 5th Mar., 1909

Date of Application (in the United Kingdom), 25th Jan., 1910

At the expiration of twelve months from the date of the first Foreign Application, the provision of Section 91 (3) (a) of the Patents and Designs Act, 1907, as to inspection of Specification, became operative

Accepted, 10th Nov., 1910

#### COMPLETE SPECIFICATION.

##### A Process for the Manufacture of Fire and Acid Proof Moulds, Crucibles and the like.

We, GEHEÜDEE SIEMENS AND COMPANY, of Lichtenberg, near Berlin, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

5 This invention relates to fire and acid proof moulds, crucibles and the like and consists in a process for manufacturing such articles in which phosphoboric acid is used, for binding together the material to be shaped. For this purpose boric acid and phosphoric acid are preferably added separately to the material, for example, anhydrous boric acid and anhydrous phosphoric acid are mixed in 10 a finely powdered condition with the material, the mixture is then rendered plastic with a suitable binding material, shaped and finally burned. It is however in many cases of considerable advantage, not to base the process upon the use of anhydrides, but to use a mixture of boric acid and common fluid phosphoric acid and this mixture is at the same time an advantageous medium for making the material which is to be shaped, plastic.

15 The process may however also be carried out in such a way, that for example, the required quantity of boric acid is immediately thoroughly mixed with the material to be worked, after which there is formed with this mixture a plastic material, with the corresponding quantity of phosphoric acid. The plastic 20 material is then shaped as desired, and dried. The phosphoboric acid forms during the drying and has the composition  $B_2O_5$ . The drying may be accomplished at a relatively low temperature in this mode of carrying out the process. It is however preferable, after the drying operation to subject the body which 25 has been made to a stronger heat, the most effective being a powerful annealing heat approximately 1000° C., in order to drive out any surplus of either of the two acids.

Specially suitable for the manufacture of moulds, crucibles or the like according to this process, is a thorough mixture of crystallised boric acid and syrup-like phosphoric acid. The products produced thereby have high refractory

[Price 8d.]



*Process for the Manufacture of Fire and Acid Proof Moulds, Crucibles and the like.*

qualities, and are even at the highest temperatures, proof against most acids; it being supposed naturally, that the principal material of which the moulds or the like are composed, has the same power of resistance as the phosphoboric acid. In order to make sure of this, the process may be carried out, so that the whole mould in its finished state, consists of only phosphoboric acid. For this purpose 5 finished phosphoboric acid is taken, and formed into the desired shape with free boric acid and phosphoric acid. For example the process may be carried out for this purpose, in that a thorough mixture of crystallised boric acid and syrup-like phosphoric acid are evaporated whilst being continuously stirred, until a 10 part of the whole mass has already changed into phosphoboric acid, the total mass still having however sufficient plasticity, so as to be capable of being shaped. The plastic mass obtained in this manner is then shaped immediately and dried. In this manner moulds or the like are obtained having a high 15 mechanical power of resistance, high refractory qualities and being exceedingly proof against acids, and moreover the porosity of such bodies is exceedingly small. The degree of porosity and especially the size of the pores can be altered within wide limits, by a suitable choice in the consistency of the plastic material, before it is shaped.

If other materials are used to form the principal part of the mould to be formed, that is to say, materials for which the phosphoboric acid only plays the 20 part of a cementitious medium, then special care must be taken, that these other materials do not form chemical changes in connection with the phosphoboric acid under conditions depending upon the purpose for which the manufactured articles are to be used. Consequently when for example the manufacture of smelting crucibles for high temperatures is dealt with, which crucibles are to 25 be cemented with phosphoboric acid, then carborundum must not be used as the principal material, because the finished body loses a great deal of solidity and power of resistance by reason of the reaction which takes place. On the other hand advantageous results are obtained, by cementing quartz powder or quartz sand with phosphoboric acid. Consequently according to the new process it is 30 possible to manufacture bodies of the highest power of resistance, the principal material of which is quartz, and which bodies are not, as has been necessary hitherto, obtained by a smelting process. The manufacture can take place at a very low temperature.

The bodies manufactured according to the present process are especially suitable for use as diaphragms or filters, because on the one hand of their high acid 35 proof qualities, and on the other hand because it is possible to obtain the finest porosity.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what 40 we claim is:—

1. A process for the manufacture of fire and acid proof moulds, crucibles and the like in which phosphoboric acid is used for binding together the material to be shaped.
2. A process according to Claim 1, in which as a preliminary process a 45 mixture of boric acid and phosphoric acid is used for rendering the mass to be shaped, plastic.
3. A mode of carrying out the process according to Claim 1, in which powdered or fine grained silicic acid (quartz, sand or the like) is made plastic with a mixture of boric acid and phosphoric acid, is then shaped, and dried.
4. A mode of carrying out the process according to Claim 2, in which phosphoboric acid is worked into a plastic material with a mixture of boric acid and phosphoric acid and is then shaped and dried, for the purpose of obtaining bodies consisting of pure phosphoboric acid.
5. A mode of carrying out the process according to Claim 4, in which a 55 mixture of boric acid and phosphoric acid whilst being stirred, is evaporated.

Process for the Manufacture of Fire and Acid Proof Moulds, Crucibles and the like.

until a plastic mass capable of being shaped is obtained which is then shaped and dried.

6. A mode of carrying out the process according to Claims 1 and 4, in which the finished body after it has been dried is subjected to an annealing heat, for the purpose of driving away any superfluity of either of the two acids.

7. The application of the process according to Claim 1 for the manufacture of diaphragms and filters.

8. Moulds, crucibles or the like, prepared according to the process or processes hereinbefore described.

10 Dated this 25th day of January, 1910.

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